Title: DES Sample Preparation: Ultrasonic Welding and Other Methods

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DES Sample Preparation: Ultrasonic Welding and Other Methods

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Overview

I. Mechanical Kneading Technique

I. Ultrasonic Welding

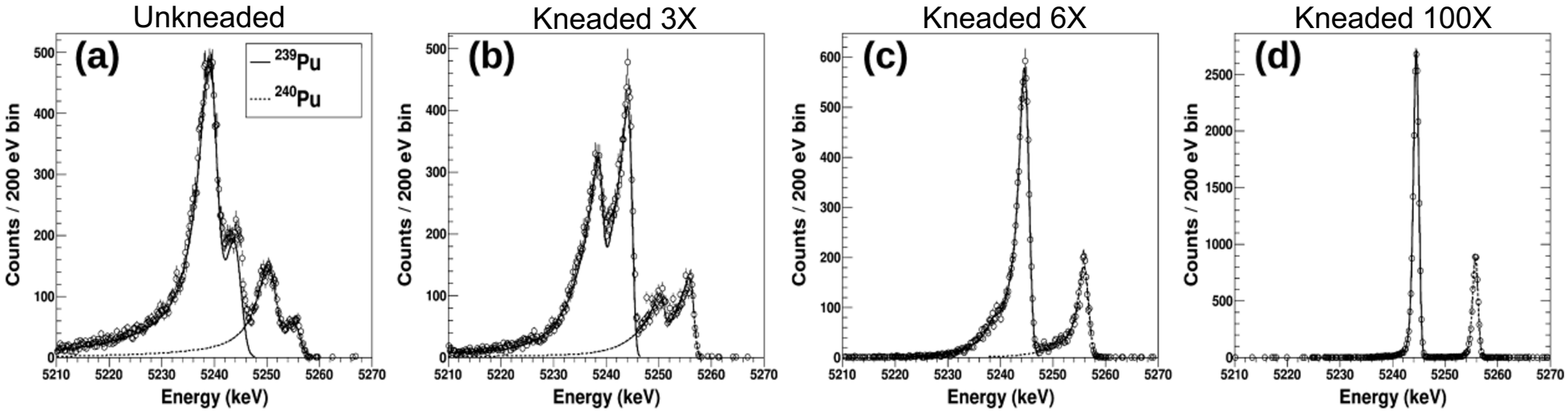
  I. Initial Small-Scale Welding Test Using Wire Bonder

  I. Ultrasonic Metal Spot Welder

I. Ultrasonic Welding Challenges and Future Work

Mechanical Kneading

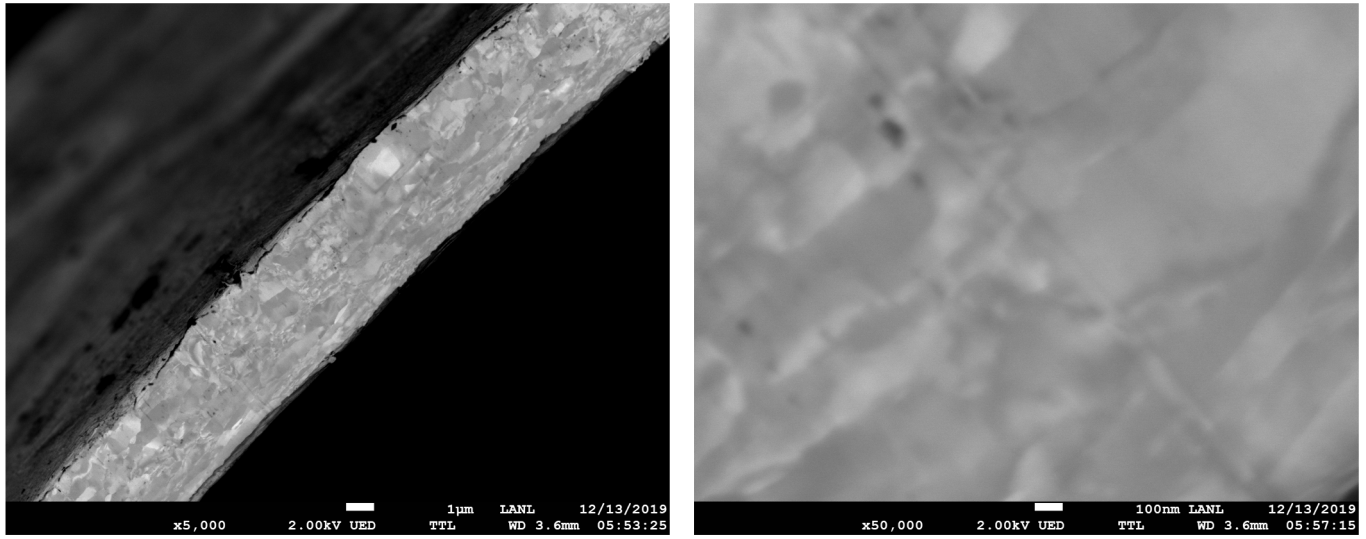
- Well-established sample preparation method that produces consistently good results (~1keV FWHM at 5MeV)
- Absorbers are squeezed between the jaws of pliers 100 times
- Breaks up crystalline residue from sample solution and homogeneously distributes it throughout absorber material to produce good energy thermalization
- Mechanical kneading effects seen in spectra:
  - counts concentrate in higher-energy peak for each isotope
  - improved resolution
  - reduced low-energy tailing



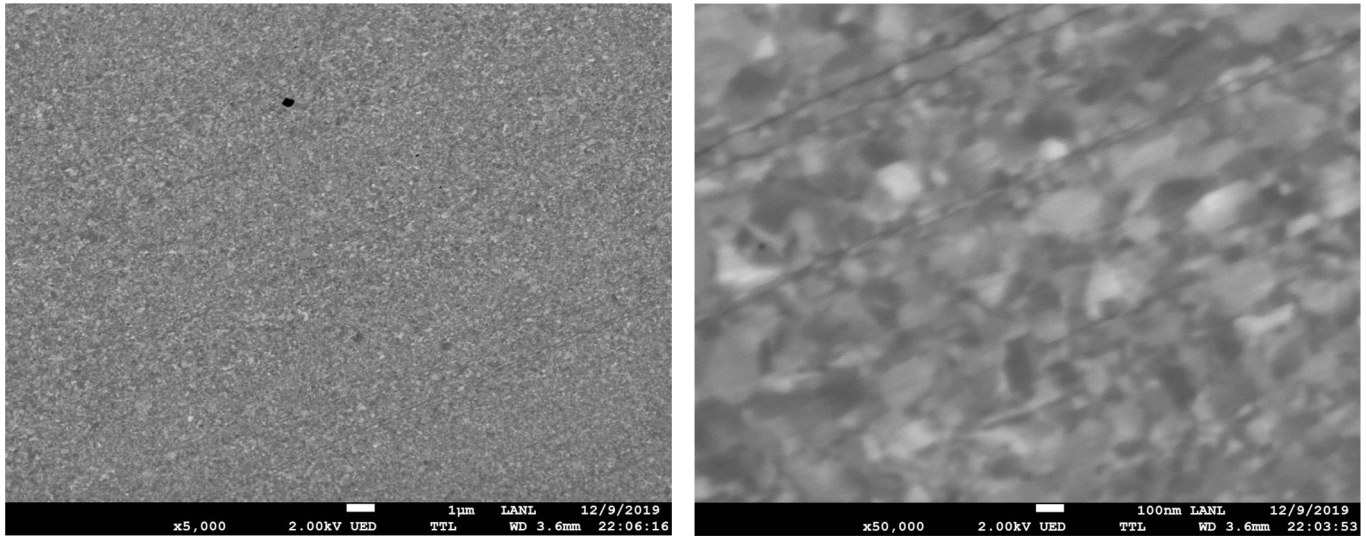
Hoover et. al., Analytical Chemistry (2015).

Mechanical Kneading

- Drawbacks:
  - Highly time-consuming
  - Extremely tedious
  - Requires great degree of skill
- Need an automated mechanical alloying process
- To provide a baseline to work towards, mechanically kneaded absorber cross-sections were characterized using SEM
- Grain structure is considered a proxy for the effectiveness of mechanical alloying



Unkneaded gold foil absorber, shows wide grain size distribution with grains as large as 1 µm. Magnification is 5000x on left and 50000x on right.



Mechanically kneaded gold foil absorber, shows a more uniform grain structure with a smaller mean size. Magnification is 5000x on left and 50000x on right.

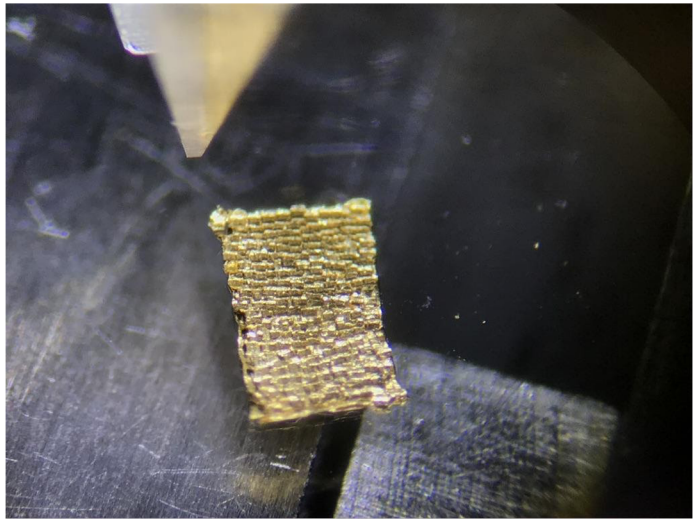
Croce, Report on Microcalorimeters for IAEA NML, (2020).

Ultrasonic Welding Overview

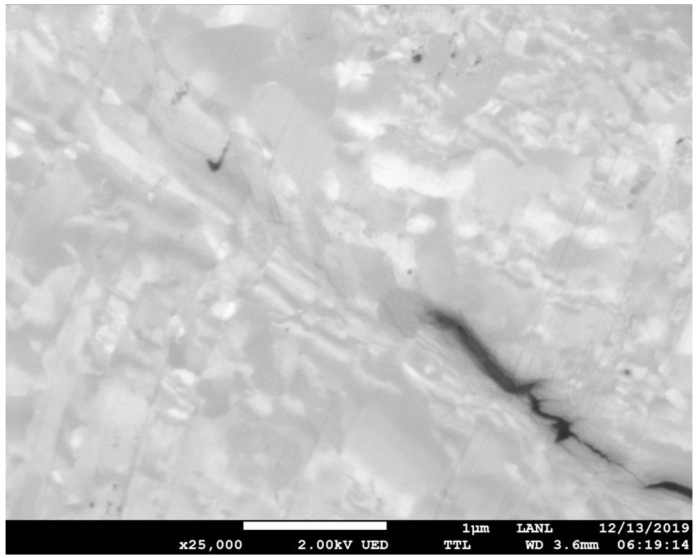
- In industry, this process is capable of fracturing oxide layers that occur on metal surfaces and incorporating the fractured oxides into the weld zone.
- Potential for incorporating nuclear material samples into a metal matrix to produce the necessary structure seen in mechanically kneaded absorbers
- Due to pandemic, delivery of Sonobond Ultrasonic Metal Spot Welder was delayed
- Initial ultrasonic welding tests were done using a manual wire bonder
  - possible to obtain sufficient ultrasonic energy density over a small area to create the desired effect

Wire Bonder as a Small-Scale Ultrasonic Welder

- West Bond 7476E manual wire bonder used to evaluate effectiveness of ultrasonic welding
  - Very small tool with relatively low power
- Gold foil absorber was folded in half with sample material inside and welded in many spots using wire bonder tool



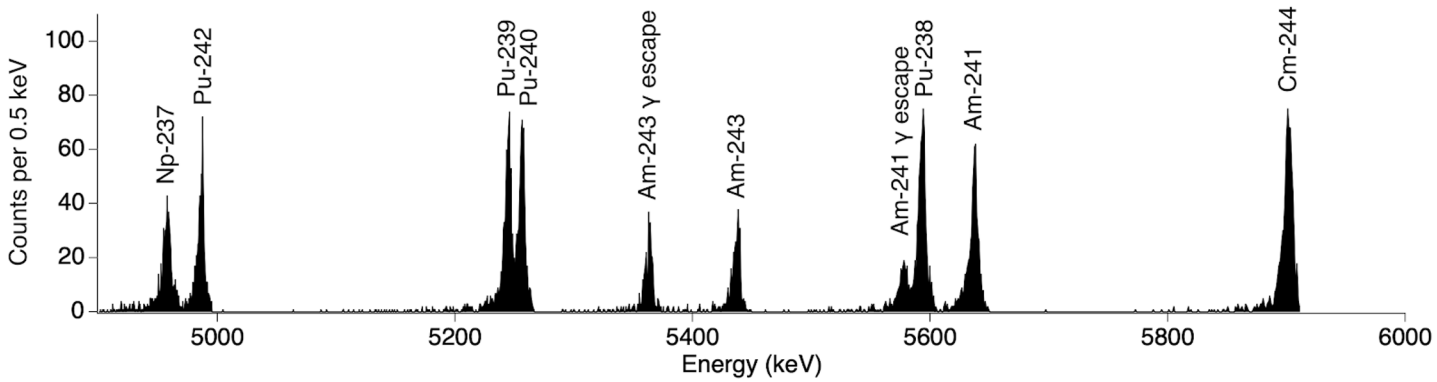
Absorber after repeated welds with the wire bonder small tool (visible in upper left).



SEM image of wire bonded absorber cross section. Grain structure that suggests plastic deformation ("flow") of the material. Magnification is 25000x.

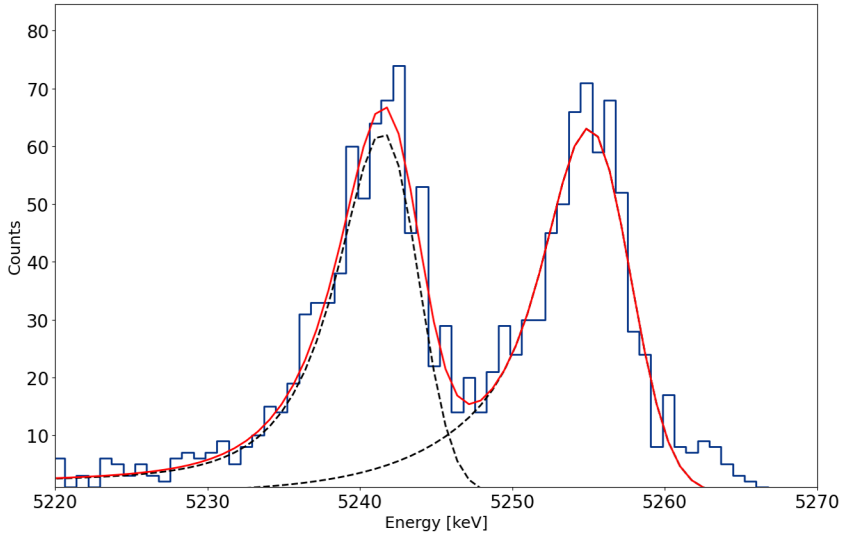
Croce, Report on Microcalorimeters for IAEA NML, (2020).

Wire Bonder as a Small-Scale Ultrasonic Welder



Mixed actinide spectrum from wire bonded absorber. All alpha-decaying nuclides known to be present in the sample are resolved.

Croce, Report on Microcalorimeters for IAEA NML, (2020).



The ^{239}Pu/^{240}Pu region is shown. Energy resolution is 4.5 keV FWHM.

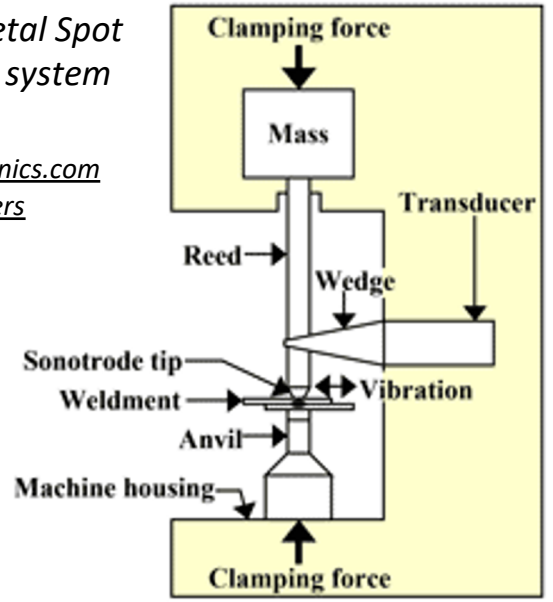
- No peak splitting as seen in unkneaded absorbers
- Achieved energy resolution and low-energy tailing is sufficient for isotopic analysis of ^{239}Pu, ^{240}Pu, and all other alpha-decaying nuclides known to be present in the sample
- Peak shapes are well described by the Bortels fitting function
- Suggests ultrasonic welding is a viable technique for the practical implementation of DES for IAEA NML samples
- Much higher ultrasonic power is needed to prepare samples in a single weld

Ultrasonic Metal Spot Welder

- Applies high frequency vibrations under high clamping force to create metallurgical bond
  - side-to-side “scrubbing” motion is capable of fracturing oxide or other material (like U or Pu samples) between two foils and incorporating into the matrix
- Machine applies too much force for formerly used gold foil absorbers
  - Gold foil was previously chosen for compatibility with mechanical kneading
- Exploring potentially better absorber materials with lower heat capacity per unit volume to allow for increase in absorber size

Sonobond Ultrasonic Metal Spot Welder and wedge-reed system diagram.

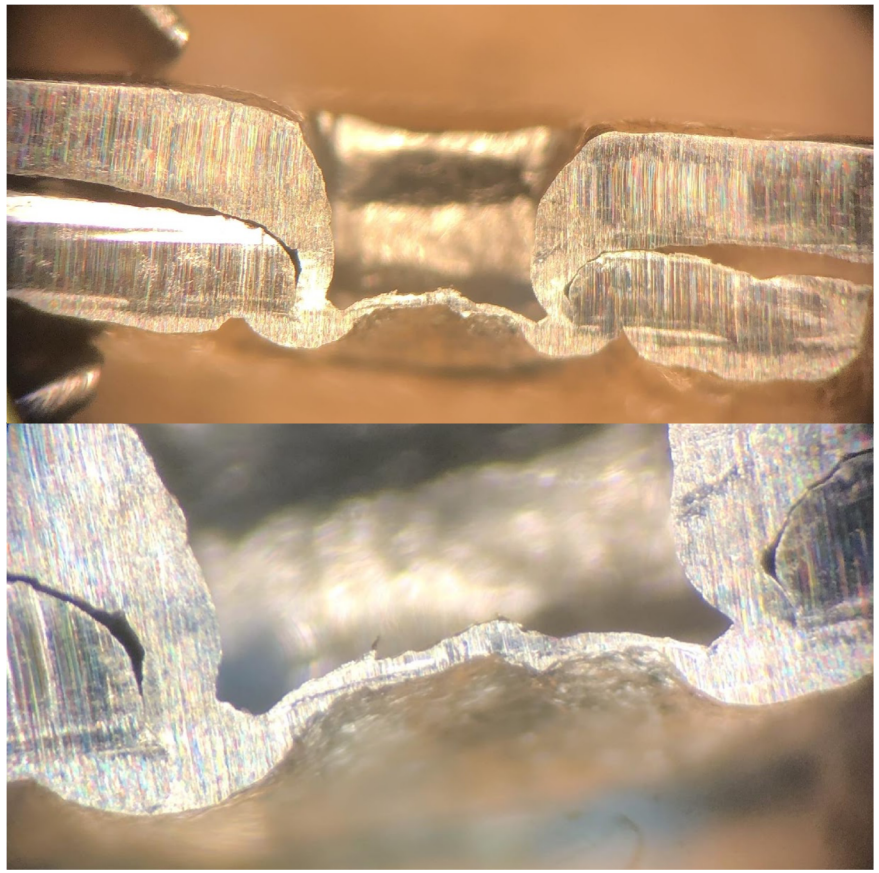
https://www.sonobondultrasonics.com/welders-bonders/metal-welders



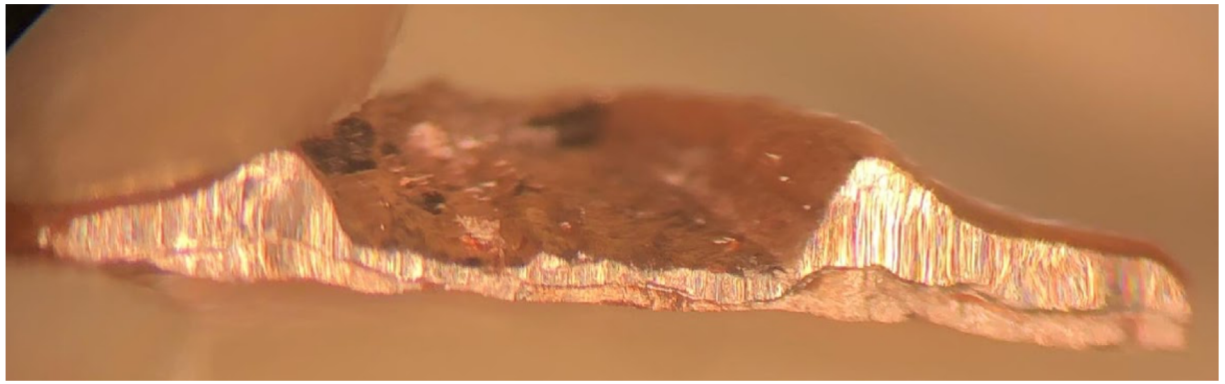
Ultrasonic Metal Spot Welder

- Alternative absorber material candidates: copper and tin
  - Tin seems to give a stronger and more consistent weld, whereas copper layers will split apart at the weld site

Dried KBr salt solution on copper and tin absorbers before folding and welding.

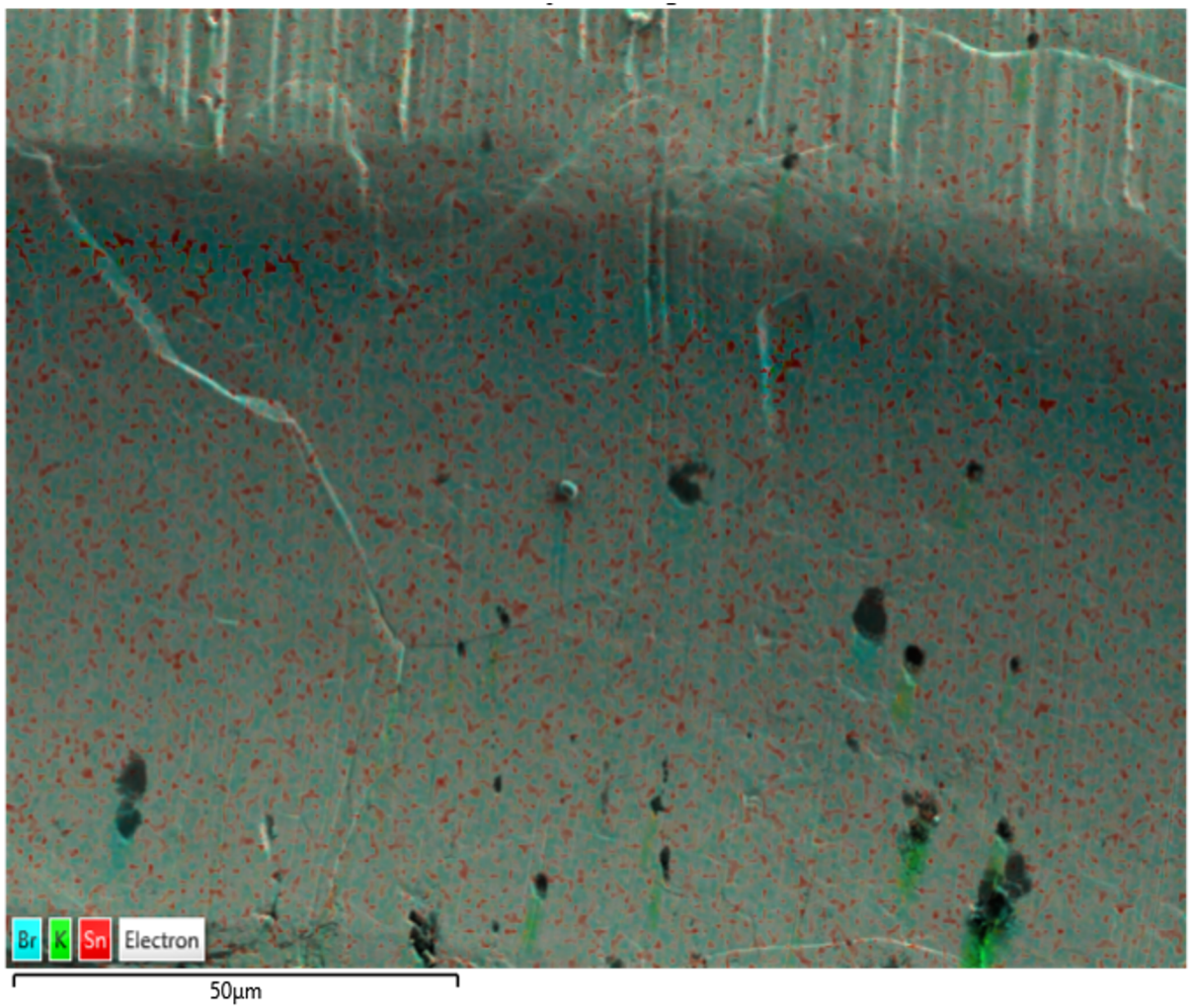


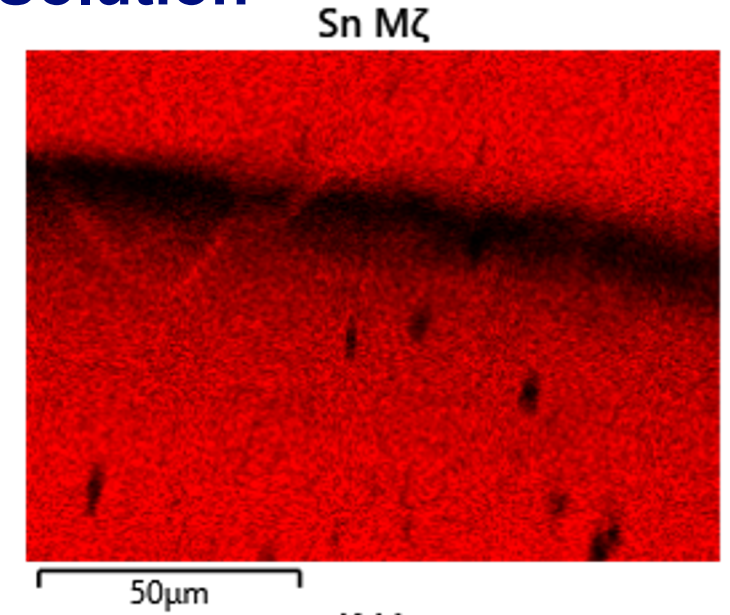
Welded tin absorber with layers completely welded together.

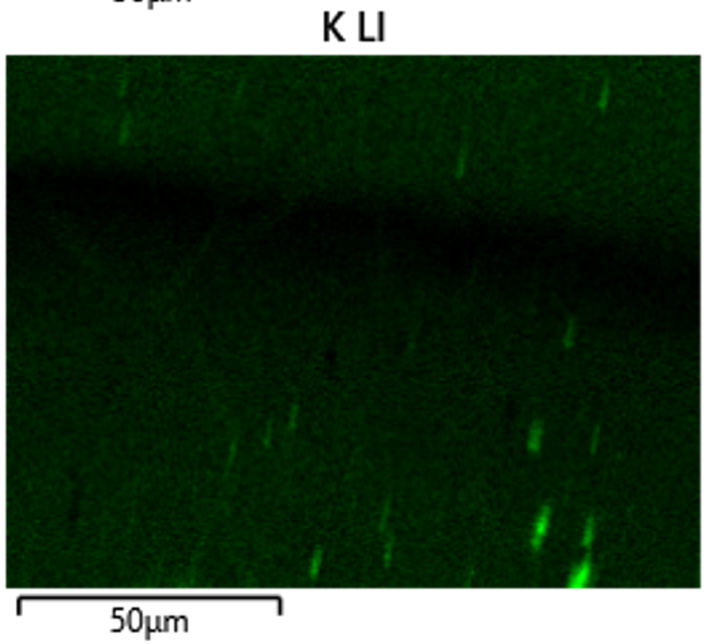


Welded copper absorber with layers still visible at the weld site.

Ultrasonic Welded Tin Absorber With Salt Solution





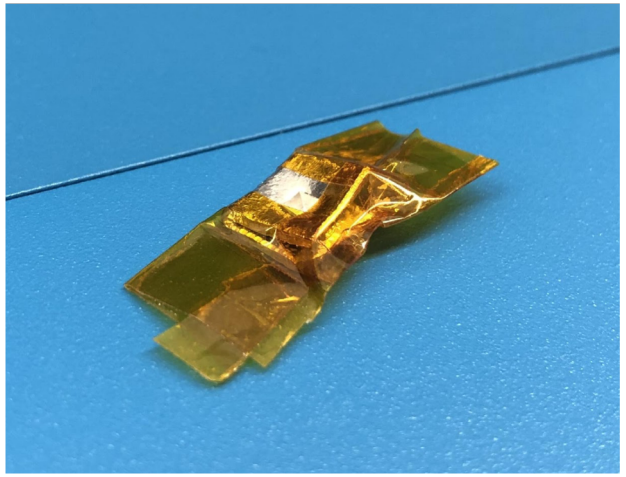


Preliminary tests using a KBr salt solution show good incorporation of sample material with absorber material.

Above: SEM image of tin absorber cross section.
Top right: EDS image showing tin on the surface.
Bottom Right: EDS image showing potassium salt deposits.

Ultrasonic Welding Challenges and Future Work

- Reaching desired absorber size
  - To reach desired heat capacity of 500pJ/K:
    - 3mm x 3mm square tin absorber needs to be approximately 1.5mm thick
    - 2.5mm x 2.5mm square tin absorber needs to be approximately 2.2 mm thick
  - Current weld tip is producing absorbers approximately 2.5mm x 2.5mm x 280μm
- Refining procedure to safely weld radioactive material
  - Vibrations cause sample material to disperse into the air during welding time
  - Consider taping around the edges of the folded absorber before welding
  - Consider wrapping and completely encapsulating folded absorber in another layer of metal before welding
    - Ensure chosen metal will not affect heat capacity of absorber if particles are incorporated in absorber during weld
  - Will the weld tip become contaminated?
    - Weld one absorber with radioactive sample, followed by an absorber with no sample
    - Measure and look for contamination on second absorber



Tin absorber with edges taped to catch any radioactive particles which may become airborne during weld.

Works Cited

1. A. S. Hoover et al., Analytical Chemistry (2015), "Measurement of the 240Pu/239Pu Mass Ratio Using a Transition-Edge Sensor Microcalorimeter for Total Decay Energy Spectroscopy" https://doi.org/10.1021/acs.analchem.5b00195.
2. M. P. Croce, “Microcalorimeters for IAEA Nuclear Material Laboratory,” September 2020, LA-UR-20-27138.
3. *Sonobond Ultrasonic Metal Welding Technology.* Sonobond, https://www.sonobondultrasonics.com/welders-bonders/metal-welders. Accessed 17 March 2021.

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